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SCHOOL OF ENGINEERING AND APPLIED SCIENCE

University of Virginia

Charlottesville, Virginia 22901

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Technical Report

MICROSTRUCTURAL INVESTIGATION OF POLYCRYSTALLINE IRON WHISKERS

By

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ABSTRACT

The microstructure of polycrystalline iron whiskers produced by chemical vapor decomposition in a magnetic field is found to consist of a core extending the length of the whiskers surrounded by a series of approximately cylindrical layers made up of particles whose diameters are about 1000 Å to 3000 Å The grain size in both the core and the surrounding particles is found to be between 50 Å to 200 Å. The very high strength of these whiskers, 800 MPa, is considered a result of extreme grain refinement strengthening.

ANGSTROMS

I. INTRODUCTION

Polycrystalline metal whiskers have received surprisingly little attention in the scientific literature, certainly much less than single crystalline whiskers, in spite of the fact that they are beginning to play an important technological role. Specifically the whiskers, made by the Schladitz process[1-3], are now being produced in quantities for industrial application[4,5].

This present paper summarizes the first results of studies on polycrystalline iron whiskers produced in this country by the Schladitz process*. The present investigation expands upon and complements earlier investigations made overseas[2,3,6,7,8].

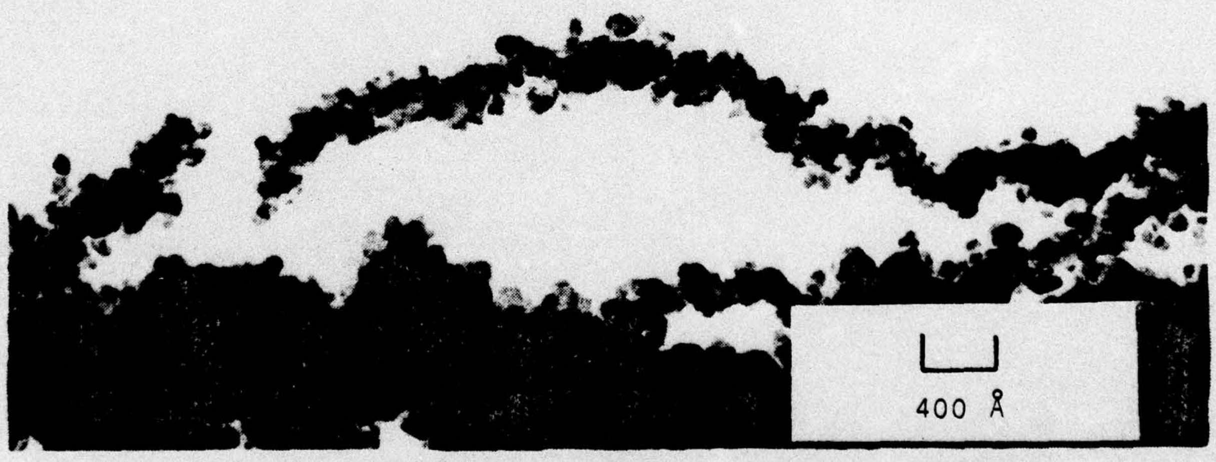

Figure I TEM micrograph of the polycrystalline core of an iron (steel) whisker showing crystal grains 50-200 Å in diameter ("primary" structure).

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*These whiskers are known in Germany as Schladitz Whiskers.

II. DESCRIPTION OF WHISKER MATERIAL

Schladitz whiskers are produced through the thermal decomposition of a metal carbonyl in the presence of a magnetic field. In the present case, iron pentacarbonyl is used. Through the use of other suitable metal carbonyls, whiskers of nickel, molybdenum, titanium and chromium have been produced[2]. A somewhat related experimental arrangement was reported by Bacigalupi[9]. As the major decomposition products of iron pentacarbonyl are iron and carbon monoxide, a significant amount of carbon is incorporated into the whiskers during their growth[6,7]. A chemical analysis performed on whiskers made in this laboratory yields a carbon content of 1.5 wt% which is consistent with the earlier findings of Dawihl and Eicke[6]. Whiskers made here were also found by neutron activation analysis to contain trace amounts of molybdenum (130 μgms/gm) and chromium (40 μgms/gm). An X-ray analysis of these whiskers will be reported in the future, the principal result[10] being that whiskers grown under "ideal" conditions consist of only α-iron and amorphous carbon; however, an oxide, Fe_3O_4, is usually present along with some Fe_3C.

Whisker diameters have been found to increase with increasing reaction time and can be varied from say 1 micron or less to 30 microns or more. The growth times are of the order of a few minutes. The length to which the whiskers can grow on the other hand has been found to be both a function of production conditions as well as the geometry of the apparatus; whiskers longer than 10 mm have been produced.

The three most outstanding characteristics of Schladitz whiskers, which make them potentially so very useful, are firstly their large, controllable surface to volume ratio[4], secondly, their high strength, which was found to be independent of whisker diameter[3], and thirdly the fact that they are amenable to a considerable variety of metallurgical treatments to modify their properties in a desired direction, e.g. increasing their corrosion resistance, high temperature strength and others[2,3,8]. Another advantage of these whiskers is the existence of a number of methods of handling the whisker to incorporate, or form them into useful macroscopic materials. One is greatly aided in this by the property that the whiskers do not lose their strength through handling.

III. MICROSTRUCTURE

The microstructural investigation conducted here on Schladitz iron (steel) whiskers is in substantial agreement with the results previously reported by Schladitz[2] and by Dawihl and Eicke[6,7] and in addition offers some substantial insights into the microstructure.

Transmission electron microscopy (TEM) of whiskers at voltages between 100 kV and 500 kV indicates that at the first stages of growth single polycrystalline filaments are formed consisting of grains 50 Å to 200 Å across. Figure 1 is a TEM micrograph which shows this primary structure.

After the core has formed, the whiskers apparently thicken without much changing their length**. In the thickening stage, a secondary structure is formed surrounding the core region. This secondary structure is made up of small particles as shown in Figure 2. A cross-section through the whiskers reveals a concentric series of approximately circular rings each made up of a large number of particles. The particles are contiguously arranged in concentric cylindrical shells so that a cross-section along the whisker axis shows particles in an array parallel to the whisker axis. In earlier experiments this concentric structure was achieved in a most obvious form through the intermittant addition of other substances, such as nickel and/or molybdenum during whisker growth, thereby demonstrating conclusively (1) that the whiskers grow through the addition of material at their surface while maintaining their symmetry and (2) the possibility of doing sophisticated metallurgy with whiskers even during their growth[2].

Upon close inspection it was found that each of these concentric layers consists of a series of essentially α-iron polycrystalline particles 1000 Å to 3000 Å in diameter arranged in a circular fashion around the core. This arrangement of particles constitutes a secondary structure with the fine grains 50 Å to 200 Å being the primary structure. In Figure 3, optical

**Special techniques have recently been devised by one of the authors (H.J.S.) which also allows growth from the tip of the whiskers.

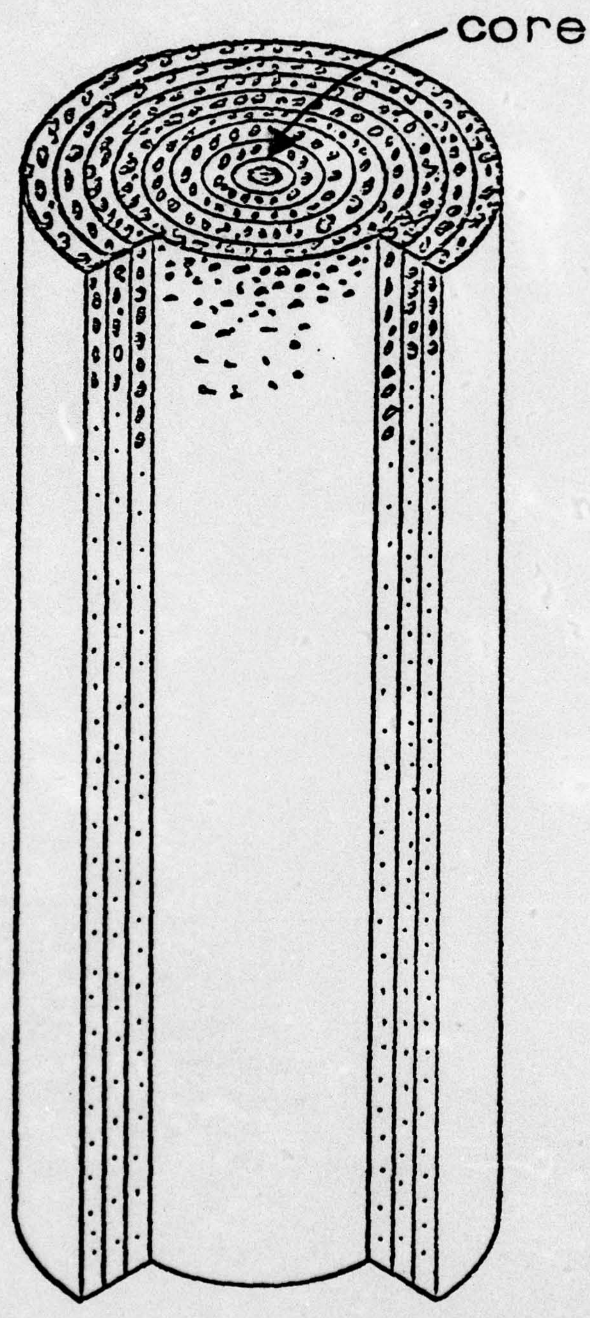

Figure 2 A schematic drawing of a cross-section through a whisker showing a series of concentric cylindrical shells made up of individual particles ("secondary" structure).

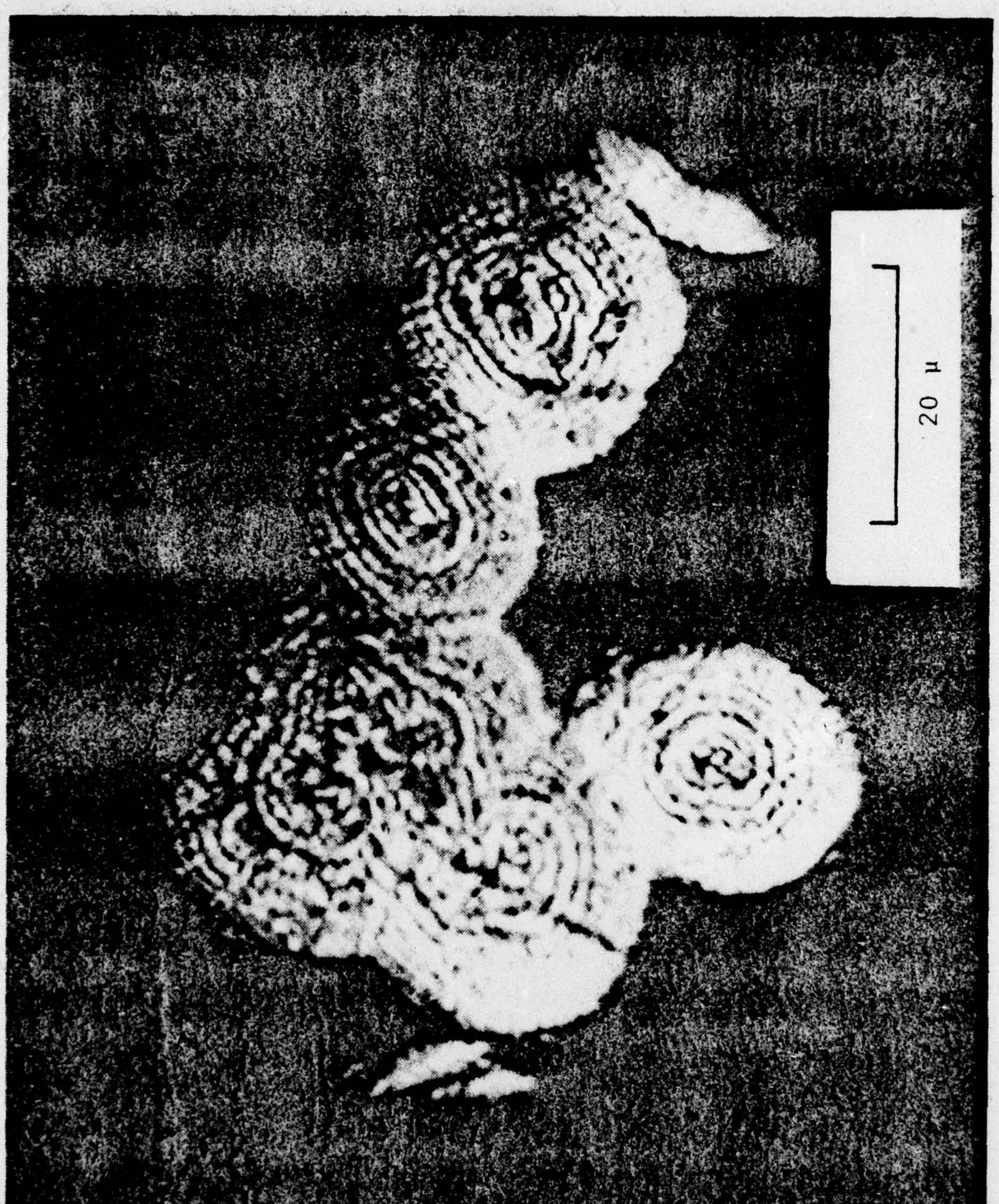

Figure 3 Optical micrograph of concentric ring structure. The specimen is an iron (steel) whisker polished and then etched with 2% NITOL.

metallographic sections of whiskers polished and etched with 2% NITOL reveal this structure quite well. These particles appear larger than 3000 Å because of diffraction effects. In Figure 4 a SEM micrograph of a similar whisker brings out the same type of structure as well as giving an accurate indication of the particle size.

Further evidence of these concentric shells of particles is shown in Figure 5 which is a TEM micrograph at 100 kV of whiskers electrochemically thinned. Whiskers grown in different parts of the reaction chamber have slightly different structural characteristics. This is probably due to slight inhomogeneities in both growth temperatures and composition throughout the reaction chamber; however, the overall secondary type of structure was found in whiskers grown throughout the chamber and was found to be independent of whisker diameter.

The whisker surface is in general not smooth, presumably due to the mentioned addition of the individual spherical particles. Beyond the minimum roughness resulting from this source, there is a surface roughness on a larger scale which is determined by the experimental growth conditions[4]. Figures 6 and 7 give examples of two such surface types found in the same batch of whiskers.

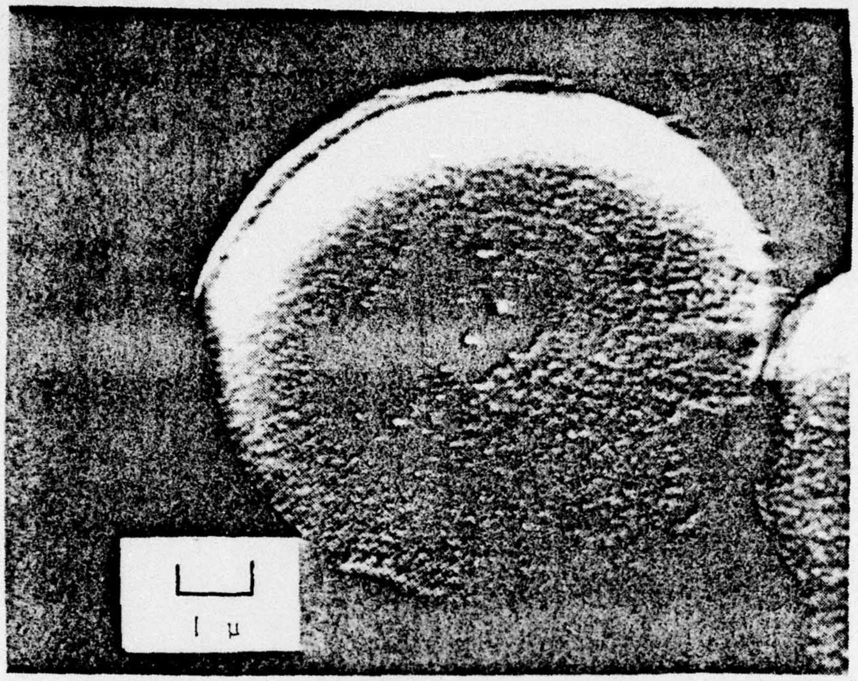

Figure 4 SEM micrograph of concentric ring structure with the rings being made up of individual particles haveing 1000-3000 Å diameter. The specimen is an iron (steel) whisker polished and then etched with 2% NITOL.

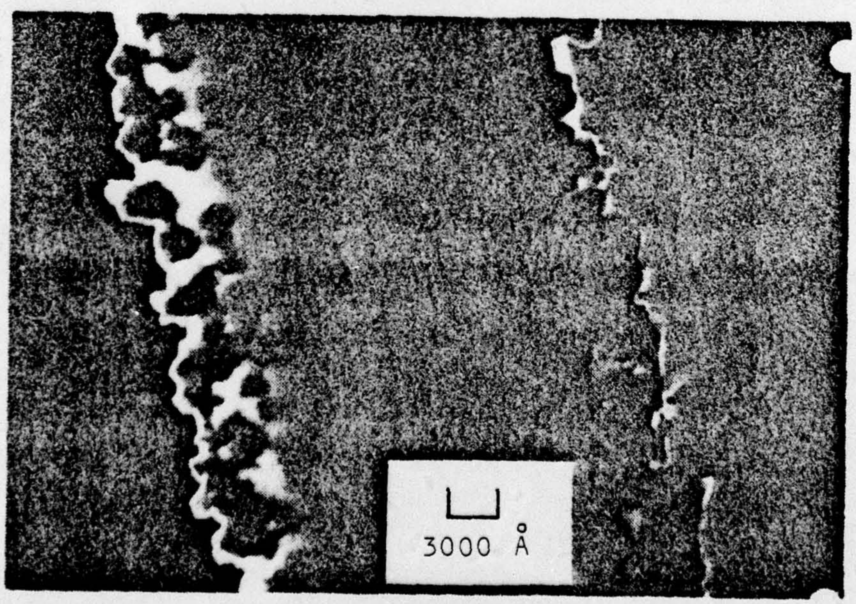

Figure 5 TEM micrograph of an electrochemically thinned iron whisker showing the concentric ring structure made up of ~ 2000 Å particles.

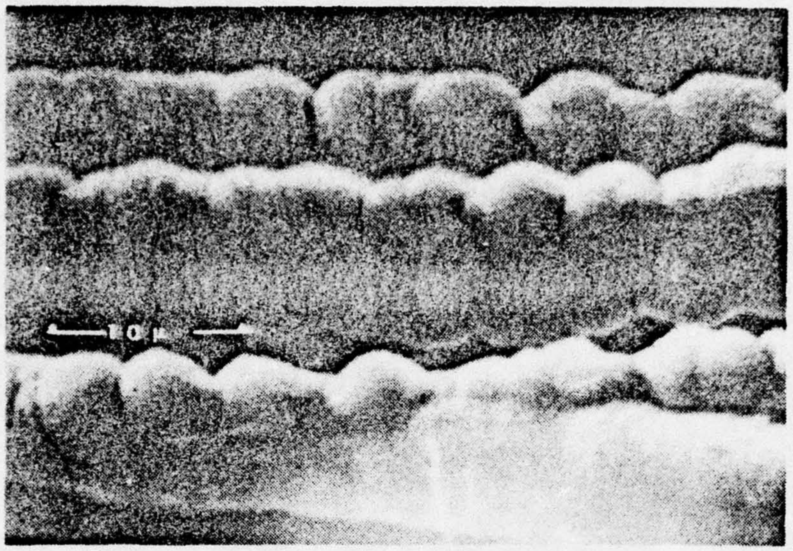

Figure 6 SEM micrograph of the surface of an iron whisker showing a particularly rough surface.

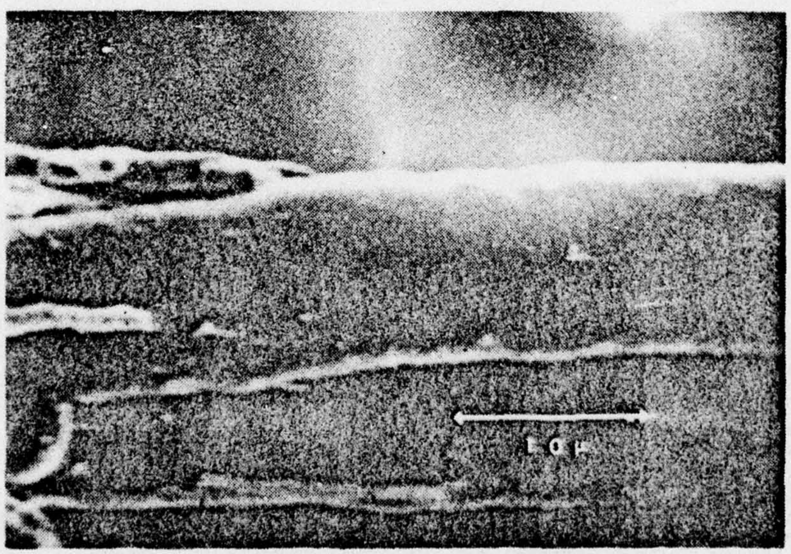

Figure 7 SEM micrograph of the surface of an iron (steel) whisker showing a more or less smooth surface.

IV. RELATION OF STRENGTH TO MICROSTRUCTURE

Characteristic of these whiskers is a very high ultimate tensile strength. The tensile strength of the whisker material has been reported to range from about 5×10^9 Pa to 8×10^9 Pa (500 to 800 Kgf/mm^2); this is about G/12 where G is the shear modulus. Individual tensile tests of whiskers performed at the University of Virginia have also yielded strengths in this range, using whiskers with diameter between 3 and 20 microns. The ultimate yield strength is found to be independent of diameter for whiskers of diameters of 3 to 20 microns.

The minimum shear stress σ to cause plastic deformation in grains of average grain diameter $\bar{d}$, present in the whiskers (50 Å to 200 Å) may be roughly estimated from the Hall-Petch equation[11,12]

$$\sigma = \sigma_o + \frac{k}{d^{\frac{1}{2}}} \qquad (1)$$

where $\sigma_o \sim 7 \times 10^7$ Pa and $k = 1.7 \times 10^7$ Pa/$mm^{\frac{1}{2}}$ are nominal values for steel[13]. With grains of 100 Å diameter, Equation (1) gives a value of 5.5×10^9 Pa (560 kgf/mm^2) for the yield strength which in this case is also the ultimate tensile stress. This calculation is therefore consistent with the very high observed strength being a result of grain refinement strengthening to the extreme limit.

SUMMARY

Two distinct microstructural regions are noted in the whiskers. These regions are firstly, a polycrystalline core or primary structure, which runs the entire length of the whiskers and secondly, a concentric series of approximately cylindrical shells composed of polycrystalline particles whose diameter is on the order of 0.1 μ. The grain size is always found to be in the neighborhood of 50 Å to 200 Å. The high strength of these whiskers is basically due to grain refinement strengthening to an extreme limit.

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REFERENCES

1. H. J. Schladitz, German Patent No. 1224934 (1964), U. S. Patents No. 3441408 and No. 3570829 (1969/1971).

2. H. J. Schladitz, Z. Metallkunde., 59, 18 (1968).

3. H. J. Schladitz, Fachberichte für Oberflächentechnik, 7, Heft 7/8 (1969).

4. H. Schladitz, Engineers' Digest, 34, June 1973, see also, Ingenieur Digest, 11, Heft 12 (1972).

5. H. J. Schladitz, Öl und Gasfeuerung, 18 164 (1973).

6. W. Dawihl and E. Eicke, Powder Metallurgy International, 3, 75 (1971).

7. W. Dawihl and E. Eicke, Berg- und Hüttenmännische Monatshefte, 116, 473 (1971).

8. H. J. Schladitz, in "Verbundwerkstoffe" (Fachberichte, Tagung Konstanz 1972, Deutsche Gesellschaft für Metallkunde., 1972), p. 153.

9. R. J. Bacigalupi, J. Appl. Phys., 34, 999 (1962).

10. Y. Katsube, personal communication.

11. E. O. Hall, Proc. Roy. Soc. (London) B64, 747 (1951).

12. H. J. Petch, J. Iron Steel Inst., 174, 25 (1953).

13. W. B. Morrison, ASM Trans. Quart. 59, 824 (1966).

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